



PERGAMON

Journal of Stored Products Research 38 (2002) 341–348

Journal of

STORED
PRODUCTS
RESEARCH

www.elsevier.com/locate/jspr

Evaluation of kaolinite-based particle films to control *Tribolium* species (Coleoptera: Tenebrionidae)[☆]

Frank H. Arthur^{a,*}, Gary J. Puterka^b

^aGrain Marketing and Production Research Center, Manhattan, KS, USA

^bAppalachian Fruit Research Station, Kearneysville, WV, USA

Accepted 6 July 2001

Abstract

Adults of *Tribolium castaneum* (Herbst), the red flour beetle, and *Tribolium confusum* (du Val), the confused flour beetle, were exposed to kaolinite-based particle film dusts. When beetles were continuously exposed to the hydrophobic particle film M-96-018 at the rate of 0.1–0.5 mg/cm², all the *T. castaneum* at 0.1 mg/cm² were dead after 3 days, but 40 ± 13.8% of the exposed *T. confusum* were still alive after 7 days. At higher concentrations, all the *T. castaneum* were dead after 2 days, but 5–6 days of exposure were needed to kill all *T. confusum*. In a subsequent test, adults of both species were exposed for 8–72 h to 0.5 mg/cm² of the particle film M-96-018, removed, then held without food for 1 week. No *T. castaneum* survived, while survival of the *T. confusum* ranged from 0 to 55 ± 17.3%, depending on the exposure interval. In a test conducted at controlled conditions of 40%, 57% and 75% r.h., 27°C, *T. confusum* were exposed for 8–72 h to the particle film M-96-018 and a hydrophilic particle film M-97-009 at the rate of 0.5 mg/cm², then removed and held either with or without wheat flour for 1 week. All the *T. confusum* exposed to the particle film M-97-009 usually survived, while survival of the *T. confusum* exposed to the particle film M-97-018 after the 1-week holding period increased with increasing relative humidity and with the presence of food. The particle film M-96-018 was effective against both the *Tribolium* species, and appears to have a potential for use in management programs to control beetles within storage facilities. Published by Elsevier Science Ltd.

Keywords: Inert dusts; Particle films; *Tribolium*; Physical controls

[☆]This paper reports the results of research only. Mention of a proprietary chemical or a trade name does not constitute a recommendation or endorsement by the US Department of Agriculture.

*Corresponding author. Tel.: +1-785-776-2783; fax: +1-785-776-2792.

E-mail address: arthur@usgmrl.ksu.edu (F.H. Arthur).

1. Introduction

Kaolinite-based particle film repellents developed by USDA-Agricultural Research Service scientists in Kearneysville, WV, USA have been used to control insect pests in fruit trees (Glenn et al., 1999). These materials are dusts that can be mixed with water and sprayed on the leaves of fruit trees to repel insects, and when used alone without water, the dusts exhibit some toxicity toward selected insect species. Kaolin is a natural mineral, and particles are manufactured and engineered with specific properties for widespread industrial use (Glenn et al., 1999).

Various new inert dusts and minerals are being tested and evaluated to control insect pests in stored raw commodities and in bulk and processed foods (Golob, 1997; Korunic, 1998). Most of the research has been with diatomaceous earth on stored grain insects, including internal feeders. The survival of stored-grain insects to diatomaceous earth can vary by species (Arthur, 2000b) and in general increasing relative humidity (r.h.) has an adverse effect on the efficacy of diatomaceous earth products (Fields and Korunic, 2000). The presence of food material can also have an adverse effect on the efficacy of inert dusts, primarily by enhancing insect survival either during or after exposure (Vrba et al., 1983; Loschiavo, 1988; Arthur, 2000c).

The kaolinite-based particle films may also have potential for use as a dry dust material in stored-product environments, especially in specialty organic markets that prohibit the use of diatomaceous earth. Several studies were conducted to evaluate the potential of kaolinite-based particle films to control *Tribolium castaneum* (Herbst), the red flour beetle, and *Tribolium confusum* (du Val), the confused flour beetle. These two beetle species are major pests inside milling and processing facilities, food warehouses, and food plants. The experiments conducted in this study were: (1) toxicity of selected particle films to *T. castaneum* and *T. confusum* exposed to different application rates or exposure intervals; (2) effects of humidity on product efficacy; and (3) effect of food on post-treatment mortality.

2. Materials and methods

2.1. Experiment 1. Effect of concentration and exposure interval on mortality of *T. castaneum* and *T. confusum*

Adults of *T. castaneum* and adult *T. confusum* were used to evaluate toxicity of a hydrophobic particle film dust, M-96-018. Bioassays were conducted in standard 100 by 15 mm² plastic Petri dishes (actual measured area of 62 cm²) lined with filter paper. For each species, six concentrations, 0 (untreated controls), 0.1, 0.2, 0.3, 0.4, and 0.5 mg/cm² were replicated 5 times (60 total dishes). The different amounts of the particle film dust were weighed into each dish and distributed around the entire area using a probe.

The insects were obtained from pesticide-susceptible laboratory colonies maintained at approximately 27°C, 57% r.h. Ten 1–2 week-old adult *T. castaneum* and *T. confusum* were put in the respective dishes for each concentration and replicate. The dishes were held on a laboratory bench at temperature conditions of approximately 23–25°C, and r.h. was not controlled or measured for this initial study. The dishes were examined daily for 7 days and beetles of both species were classified as alive or dead. The criteria for mortality was no visible movement when

prodded with a probe. The data were analyzed using the ANOVA procedure of the Statistical Analysis System (SAS Institute, 1987) with species and concentration as main effects and insect survival as the response variable.

2.2. Experiment 2. Effect of exposure interval on delayed mortality of *T. castaneum* and *T. confusum*

This test was also conducted in the laboratory at temperatures of approximately 23–25°C, using insects from the pesticide-susceptible laboratory colonies. Adults of both species were exposed for 8, 16, 24, 48, and 72 h to 0.5 mg/cm² of the particle film M-96-018, which is the labeled application rate for the Protect-It™ formulation of diatomaceous earth. For each species, there were two sets of four replicates at each exposure interval plus an untreated control dish (100 in total). The particle film was weighed into the Petri dish and distributed as described above. For each species, 10 1–2 week-old adults of each species were exposed in each dish. After the exposure interval, all living and dead beetles were transferred to new Petri dishes lined with filter paper and held for 1 week without food, as for previous tests with the pyrethroid cyfluthrin (Arthur, 1998a, b) or diatomaceous earth (Arthur, 2000b). Survival was assessed again after this one-week holding period, and data were analyzed using the SAS ANOVA procedure with species and exposure interval as main effects.

2.3. Experiment 3. Effect of temperature and relative humidity on mortality of *T. confusum* exposed to particle films M-96-018 and M-97-009

The first two experiments indicated that *T. castaneum* was much more susceptible to the particle film than *T. confusum*. Therefore, only *T. confusum* from the laboratory colony was exposed as in Experiment 2 to particle film M-96-018 at 27°C; 40%, 57%, and 75% r.h., then held either with or without food. Humidity chambers were created by lining three 26 by 36.5 by 15 cm³ plastic boxes with waffle-type grids in the bottom, and pouring 750 ml of either saturated K₂CO₃, NaBr, or NaCl to the level of the grid. These specific salts maintained humidities of 40%, 57%, and 75%, respectively (Greenspan, 1977).

At each temperature–humidity combination, there were two sets of four replicates plus an untreated control for each interval (100 dishes for each r.h.), and each humidity assay was done in random order on successive weeks. M-96-018 and 10 1–2 week old *T. confusum* adults were added to each dish as described previously. Upon completion of the exposure intervals, beetles from both sets of dishes were classified as alive or dead, then removed and placed either in clean dishes or dishes that contained 100 mg wheat flour (dishes also lined with filter paper). These dishes were placed in the humidity chambers where the beetles had been exposed, put back in the temperature incubator, and held for an additional week. Survival was then assessed again and the beetles discarded. After this test, the same procedures were used for a different particle film, the hydrophilic film M-96-009. Data for both particle films were analyzed using the SAS ANOVA procedure with r.h., exposure interval, and the presence of flour as main effects and the initial and 1-week mortalities as repeated measures.

3. Results

3.1. Experiment 1. Effect of concentration and exposure interval on mortality of *T. castaneum* and *T. confusum*

Data for controls of each species were not analyzed because all the beetles on each of the daily observations survived. Main effects species ($F = 1267.9$, $df = 1, 40$), concentration ($F = 53.1$, $df = 4, 40$), the repeated measure day ($F = 393.9$, $df = 6, 240$) and all interactions were significant ($P < 0.05$) for the percentage of adult survival.

T. castaneum was much more susceptible than *T. confusum* to the particle film M-96-018 (Fig. 1). At the rate of 0.1 mg/cm^2 , all *T. castaneum* were dead after 3 days, but $40 \pm 13.8\%$ of the exposed *T. confusum* were still alive after 7 days of continuous exposure (Fig. 1A). At concentrations of 0.3 mg/cm^2 or greater, all *T. castaneum* were dead after 2 days, while 5–6 days were required to kill *T. confusum* (Fig. 1C–E). Although survival of *T. confusum* progressively decreased with increases in concentration and exposure interval, its survival was higher than *T. castaneum* ($P < 0.05$) for all concentrations at 1–4 days of exposure.

3.2. Experiment 2. Effect of exposure interval on delayed mortality of *T. castaneum* and *T. confusum*

Survival of both species in the controls after initial exposures and after the 1-week holding period was usually 100%. Corrections for mortality when necessary were done using Abbotts' formula (Abbott, 1925). This study again showed *T. castaneum* was much more susceptible than *T. confusum* to the particle film M-96-018. Main effects species ($F = 36.9$, $df = 1, 30$), exposure interval ($F = 8.5$, $df = 4, 30$) and the species by time interaction were significant ($P < 0.05$) for survival 1 week after the beetles were removed from the exposure interval. No *T. castaneum* survived, while survival of *T. confusum* ranged from $55 \pm 17.3\%$ when exposed for 8 h to 0 when exposed for 72 h (Fig. 2). Comparisons at 8–24 h were significant ($P < 0.05$) for survival between the 2 species.

3.3. Experiment 3. Effect of temperature and relative humidity on mortality of *T. confusum* exposed to particle films M-96-018 and M-97-009

All *T. confusum* in the controls of both tests and when exposed to the hydrophilic particle film M-97-009 survived at all exposure intervals and r.h.s regardless of whether the beetles were held with or without food. In the test with particle film M-96-018, all untreated controls were alive after the initial exposures and after the 1-week holding period. In addition, all *T. confusum* were alive upon completion of the exposure interval to both particle films at all r.h.s. However, after the 1-week holding period, survival of beetles exposed to particle film M-96-018 was significant ($P < 0.05$) for main effects presence of the flour food source during the 1-week holding period ($F = 110.0$, $df = 1, 90$), r.h. ($F = 32.1$, $df = 2, 90$), original exposure interval ($F = 107.1$, $df = 4, 90$) and all interactions.

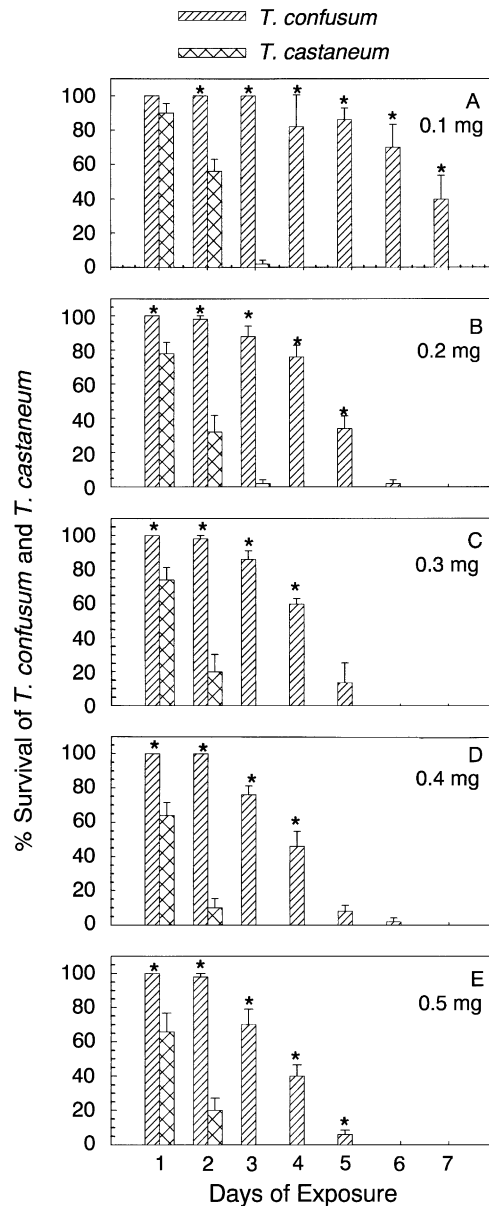


Fig. 1. Survival of *T. castaneum* and *T. confusum* exposed for 1–7 days to 0.1–0.5 mg/cm² hydrophobic particle film M-97-0018. Means marked with an asterisk are significantly different ($P < 0.05$).

Survival of the *T. confusum* exposed for 8, 16 and 24 h to the particle film M-96-018 was the same ($P \geq 0.05$) regardless of whether beetles were provided with flour during the 1-week holding period. However, when beetles were exposed for 48 and 72 h at 40% and 57% r.h., the presence of flour led to a significant increase in survival (Fig. 3A and B). A humidity effect similar to that seen with diatomaceous earth was also evident because

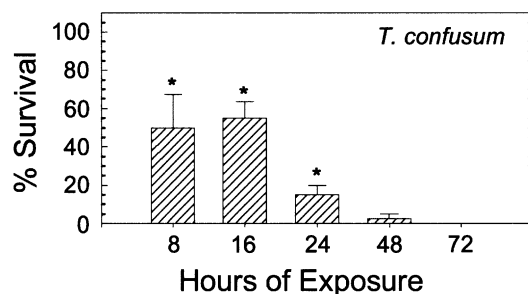


Fig. 2. Survival of *T. confusum* 1 week after exposure to 0.5 mg/cm² hydrophobic particle film M-97-0018 for 8–72 h. Means marked with an asterisk are significantly different ($P < 0.05$). Data for *T. castaneum* are not shown because no insects survived exposure.

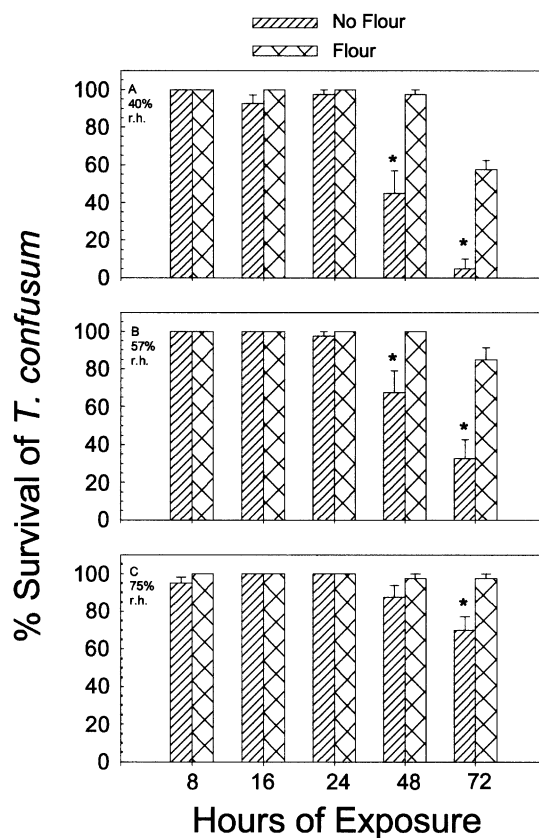


Fig. 3. Survival of *T. confusum* after exposure for 8–72 h to 0.5 mg/cm² hydrophobic particle film M-97-0018. Tests were conducted at 40%, 57%, and 75% r.h., and beetles were held either with or without flour after they were exposed. Means for exposure on flour versus exposure without flour marked with an asterisk are significantly different ($P < 0.05$).

as r.h. increased from 40% to 57% and then to 75%, survival after exposure at 48 and 72 h also increased.

4. Discussion

The discrepancy between the relative susceptibility of *T. castaneum* and *T. confusum* was higher for the particle film M-96-018 than in tests with the diatomaceous earth Protect-It® (Arthur, 2000b). Two to 3 days of exposure to 0.5 mg/cm² of diatomaceous earth were needed to kill all the *T. castaneum*, but in this test exposure to 0.1 mg/cm² of the particle film M-96-018 had the same effect. The 0.5 mg/cm² rate of the particle film M-96-018 killed all beetles after 8 h of exposure. Results for *T. confusum* exposed to the particle film M-96-018 were more comparable to those of diatomaceous earth. In tests with pyrethroid insecticides, *T. castaneum* was more tolerant to cyfluthrin than *T. confusum* (Arthur, 1998a, b), but results were reversed for deltamethrin dust (Arthur, 1997).

Survival of *T. castaneum* and *T. confusum* after exposure to particle film M-96-018 increased with increasing r.h., similar to effects with diatomaceous earth (Arthur, 2000b). Inert dusts such as diatomaceous earth disrupt the absorption of lipids through the epicuticle, and insects become vulnerable to desiccation (Glenn et al., 1999). Diatomaceous earth can be abrasive, causing additional damage to the cuticle (Korunic, 1997), and while the particle film may be less abrasive, its effects on the cuticle are similar. When plant surfaces are treated with the particle film M-96-018 (Glenn et al., 1999), the material acts primarily as a protective barrier but once the insects become coated with the film, different physiological effects may be produced. The film could inhibit breathing through plugging of the spiracles and cause water loss through damage to the cuticle. These effects would be mitigated as r.h. increases. Survival of the *T. confusum* held without food was greater where humidity was controlled (Experiment 3) than when it was not (Experiment 2). Some of the differences in results between Experiments 1 and 2 may also be explained by variations and fluctuations in the humidity.

When *T. castaneum* and *T. confusum* were given food after exposure to the particle film M-96-018, their survival greatly increased, as in studies on these species with diatomaceous earth (Arthur, 2000b) or cyfluthrin (Arthur, 1998c). Although survival can increase if insects are given food after exposure to conventional insecticides (Arthur, 1998c), the effects may be more pronounced when combined with inert dusts (Arthur, 2000a, c). Exposed insects may also use the food to mechanically clean and remove the dusts through physical abrasions, thus sanitation can be extremely important in insect control when using inert dusts. Although it may be impossible to completely remove all food and refuse, targeted areas should be managed and kept clean to maximize the effectiveness of insecticidal dusts.

T. castaneum appeared to be very susceptible to the particle film M-96-018, compared to the diatomaceous earth Protect-It®; however, susceptibility of *T. confusum* to both products appeared to be similar. Commercial formulations of diatomaceous earth vary in toxicity (Fields and Korunic, 2000), and therefore it is difficult to compare kaolinite-based particle films to all commercial DE products. However, many diatomaceous earths contain either amorphous or crystalline silica, which can preclude use in some organic markets. In addition, we evaluated the particle film only as a surface treatment, and further research is necessary to determine its potential as a grain treatment.

Acknowledgements

We thank C.K. Hoernemann for excellent technical assistance with the research, and Englehardt Corporation for product and financial support. We also thank J.J. Knodel and Dr. J.L. Zettler for reviewing the paper prior to journal submission.

References

- Abbott, W.S., 1925. A method of computing the effectiveness of an insecticide. *Journal of Economic Entomology* 18, 265–267.
- Arthur, F.H., 1997. Differential effectiveness of deltamethrin dust on wood, concrete, and tile surfaces against three stored-product beetles. *Journal of Stored Products Research* 33, 167–173.
- Arthur, F.H., 1998a. Residual toxicity of cyfluthrin wettable powder against *Tribolium confusum* exposed for short time intervals on concrete. *Journal of Stored Products Research* 34, 19–25.
- Arthur, F.H., 1998b. Residual studies with cyfluthrin wettable powder: toxicity towards red flour beetles (Coleoptera: Tenebrionidae) exposed for short time intervals on treated concrete. *Journal of Economic Entomology* 91, 309–319.
- Arthur, F.H., 1998c. Effects of a flour food source on red flour beetle (Coleoptera: Tenebrionidae) survival after exposure on concrete treated with cyfluthrin. *Journal of Economic Entomology* 91, 773–778.
- Arthur, F.H., 2000a. Impact of accumulated food on survival of *Tribolium castaneum* on concrete treated with cyfluthrin wettable powder. *Journal of Stored Products Research* 36, 15–23.
- Arthur, F.H., 2000b. Toxicity of diatomaceous earth to red flour beetles and confused flour beetles: effects of temperature and relative humidity. *Journal of Economic Entomology* 93, 526–532.
- Arthur, F.H., 2000c. Impact of food source on survival of red flour beetles and confused flour beetles (Coleoptera: Tenebrionidae) exposed to diatomaceous earth. *Journal of Economic Entomology* 93, 1347–1356.
- Fields, P., Korunic, Z., 2000. The effect of grain moisture content and temperature on the efficacy of diatomaceous earths from different geographical locations against stored-product beetles. *Journal of Stored Products Research* 36, 1–13.
- Glenn, D.M., Puterka, G.J., Vanderwet, T., Byers, R.E., Feldhake, C., 1999. Hydrophobic particle films, a new paradigm for suppression of arthropod pests and plant diseases. *Journal of Economic Entomology* 92, 759–771.
- Golob, P., 1997. Current status and future perspectives for inert dusts for control of stored product insects. *Journal of Stored Products Research* 33, 69–79.
- Greenspan, L., 1977. Humidity fixed points of binary saturated aqueous solutions. *Journal of Research of the National Bureau of Standards—Physics and Chemistry* 81A, 89–96.
- Korunic, Z., 1997. Rapid assessment of the insecticidal value of diatomaceous earths without conducting bioassays. *Journal of Stored Products Research* 33, 219–229.
- Korunic, Z., 1998. Diatomaceous earths, a group of natural insecticides. *Journal of Stored Products Research* 34, 87–97.
- Loschiavo, S.R., 1988. Availability of food as a factor in the effectiveness of a silica aerogel against the merchant grain beetle (Coleoptera: Cucujidae). *Journal of Economic Entomology* 81, 1237–1240.
- SAS Institute, 1987. SAS/STAT Guide for Personal Computers, 6th Edition. Statistical Analysis System Institute, Cary, NC.
- Vrba, C.H., Arai, H.P., Nosal, M., 1983. The effect of silica aerogel on the mortality of *Tribolium confusum* (du Val) as a function of exposure time and food deprivation. *Canadian Journal of Zoology* 61, 1481–1486.